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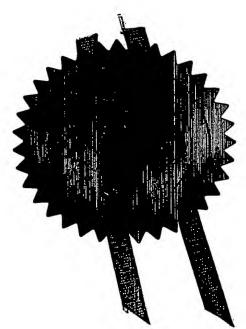
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- 2 OCT 2003

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1.	Your Reference		<b>2</b> OCT
2.	Application number	0323067.9 020CT03 E841620-1 D02B46-	
3.	Full name, address and postcode of the or each Applicant Country/state of incorporation (if applicable)	P01/7700 0.00-0323067.9  Advanced Gel Technology Limited Unit 41 Campus Road Listerhills Science Park Bradford BD7 1HR 835 299900 \ Incorporated in: United Kingdom	· · · · · · · · · · · · · · · · · · ·
4.	Title of the invention	VISCOUS FLUIDS	<del></del> .
5.	Name of agent	APPLEYARD LEES	1
	Address for service in the UK to which all correspondence should be sent	o 15 CLARE ROAD HALIFAX HX1 2HY	
•	Patents ADP number	190001	<del></del>
<b>6</b> .	Priority claimed to:	Country Application number Date of filing	
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#### VISCOUS FLUIDS

This invention relates to viscous fluids and particularly, although not exclusively, relates to the reduction of the viscosity of viscous fluids to facilitate their flow between two locations. Preferred embodiments relate to the reduction of the viscosity of viscous petroleum to facilitate its transport between a place where it is produced and a point downstream thereof.

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There are many known petroleum-containing formations from which very little petroleum can be obtained by normal production techniques because the petroleum viscosity is so high that the petroleum will not flow at formation conditions even if a substantial pressure differential, either natural or artificially induced as by injecting water or other fluids into the formation, is applied to the petroleum. These petroleum formations are sometimes referred to as heavy oil formations, and for the purpose of this disclosure, by heavy oil or viscous petroleum it is meant crude petroleum having an API gravity less than about 25° AFI at 75°F.

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Various techniques have been disclosed for stimulating the recovery of viscous petroleum or heavy oil facilitating its transport along pipelines from production facility to a refinery. However, there still exists a problem, especially where the petroleum extremely viscous such as that found in tar sand deposits.

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It is an object of the present invention to address problems associated with the flow and/or transport of viscous fluids.

According to a first aspect of the invention, there is provided a method of reducing the viscosity of a viscous composition which is arranged to flow along a fluid flow path, said method comprising contacting the viscous composition with a treatment fluid formulation, said treatment fluid formulation comprising a third polymeric material which comprises a second polymeric material cross-linked by a first polymeric material, wherein said first polymeric material comprises:

(i) a first polymeric material having a repeat unit of formula

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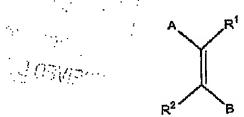
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wherein A and B are the same or different, are selected from optionally-substituted aromatic and heteroaromatic groups and at least one comprises a relatively polar atom or group and  $R^{\rm I}$  and  $R^{\rm 2}$  independently comprise relatively non-polar atoms or groups; or

(ii) a first polymeric material prepared or preparable by providing a compound of general formula

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wherein A, B,  $R^1$  and  $R^2$  are as described above, in an aqueous solvent and causing the groups C=C in said compound to react with one another to form said first polymeric material.

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The ratio of the viscosity of the viscous composition immediately prior to contact with the treatment fluid formulation to the viscosity after contact with the treatment fluid formulation is preferably at least 1.2, more preferably at least 1.5.

The viscosity of the viscous composition after contact with the treatment fluid formulation is preferably less than 300cp, more preferably less than 200cp, especially less than 100cp measured at 25°C and 1000s<sup>-1</sup>.

Whilst the method may be used to reduce the viscosity of many types of viscous compositions provided that the viscous compositions can be caused to form an emulsion when contacted with said treatment fluid formulation, it is preferred that said viscous composition comprises petroleum.

said viscous composition may be derived from tar sand deposits. It may be derived from a deep well wherein the composition may be sufficiently warm down the well to enable it to flow; but the viscosity rises as the composition is withdrawn from the well (and cools) making it more difficult to flow.

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Said treatment fluid formulation is preferably not injected into an injection well of a subterranean formulation in order to contact said viscous composition.

Said treatment fluid formulation is preferably initially contacted with said viscous composition at or downstream of a production means, for example a producing face of a subterranean formation.

Said treatment fluid formulation is preferably not used to drive the viscous composition through a subterranean formation.

Said fluid flow path is preferably defined by a conduit means.

Said conduit means preferably includes a first conduit part (e.g. a pipeline) which is arranged downstream of a production means, preferably above ground level. Said first conduit part preferably contains said viscous composition after contact with the treatment fluid formulation.

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Said first conduit part may be circular in cross-section. Said part may have a cross-sectional area for at least part of its length of at least 5cm, preferably at least 10cm. In some cases, the cross-section may have a diameter of up to 2m. Said first conduit part may have a length of at least 5m, preferably at least 20m, especially at least 50m. In some cases, said first conduit part may have a length of thousands of metres. Long pipes may be arranged to deliver petroleum to a refinery; such pipes may extend partly above and partly below ground.

Said fluid flow path (e.g. said conduit means) may extend between a first point, above ground (e.g. a refinery) and a second point below ground. Said second point may be the producing face of a subterranean formation.

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Said fluid flow path may be defined, in part, by a second conduit part which extends upwardly from below ground to above ground. Said second conduit part may be a riser pipe. Said second conduit part preferably contains said viscous composition after contact with the treatment fluid formulation.

said treatment fluid formulation is preferably arranged to emulsify said viscous composition on contact therewith. Preferably, flow along said fluid flow path is turbulent, at least in part, thereby to facilitate formation of said emulsion. Preferably, flow is turbulent at the point of initial contact of said viscous composition with said treatment fluid formulation so that said composition is emulsified on contact with said formulation.

In the method, a delivery flow path is preferably defined which is arranged to communicate with said fluid flow path wherein said treatment fluid formulation is dosed into said viscous composition in said fluid flow path via said delivery flow path. Said delivery flow path preferably communicates with said fluid flow path at or downstream of a producing face of the subterranean formation.

The ratio of the flow rate (in weight per unit time) of treatment fluid formulation in said delivery flow path to the flow rate (in the same units) of viscous composition in said fluid flow path may be in the range 0.2 to 2,

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preferably in the range 0.2 to 1, more preferably in the range 0.4 to 0.8, especially in the range 0.6 to 0.7.

Suitably, the ratio of the wt% of said viscous composition to the wt% of said treatment fluid formulation contacted with the viscous composition over a period of one minute, is at least 0.8. is preferably at least 1.0, is more preferably at least 1.2, and especially is at least 1.4. The ratio may be less than 4, suitably less than 3, preferably less than 2.5, more preferably less than 2, especially less than 1.8.

Preferably, immediately after contact between said viscous composition and said treatment fluid formulation, the composition in said fluid flow path includes 30 to 80 wt% (preferably 40 to 80wt%, more preferably 50 to 70 wt%) of material derived from said viscous composition and 20 to 70wt%, (preferably 20 to 60wt%, more preferably 30 to 50wt%, especially 30 to 45wt%) of material derived from said treatment fluid formulation.

Suitably, immediately after contact between said viscous composition and said treatment fluid formulation, the composition in said fluid flow path includes at least 20wt%, preferably at least 25wt%, more preferably at least 30wt%, water; and at least 40wt%, preferably at least 50wt%, more preferably at least 50wt% of said viscous composition, especially of oil.

The amount of water in the composition in said fluid flow path immediately after contact between said viscous composition and said treatment fluid formulation is preferably less than 70wt%, more preferably less than

60wt%, especially less than 50wt%, more preferably 40wt% or less. The amount of water may be in the range 20 to 50wt%.

5 Preferably, prior to the treatment fluid formulation contacting the viscous composition, it has attained at least 70% of the maximum viscosity attainable for the formulation at the temperature at which it is to contact the viscous composition. Preferably, it has attained at least 80%, more preferably 90%, especially about 100% of its maximum viscosity. Thus, in the especially preferred embodiment, said first and second polymeric materials are substantially completely reacted to form said third polymeric material prior to contact with said viscous composition.

Said treatment fluid formulation preferably has a viscosity at 25°C and 1000s<sup>-1</sup> of greater than 20cp and, preferably, not greater than 50cp.

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Said treatment fluid formulation is preferably aqueous. It may include at least 70wt%, preferably at least 80wt%, more preferably at least 90wt%, especially at least 95wt% water. The amount of water may be less than 99.6wt%, preferably less than 99wt%.

Said treatment fluid formulation suitably includes at least 0.2wt%, preferably at least 0.4wt%, especially at least 0.5wt% of said third polymeric material. Said formulation preferably includes less than 10wt%, more preferably less than 8wt%, especially less than 5.5wt% of said third polymeric material.

In a preferred embodiment, said treatment fluid formulation includes 94.5 to 99.6wt% water and 0.4 to 5.5. wt% of said third polymeric material; and the ratio of the wt% of said treatment fluid formulation to the wt% of said viscous composition contacted in the method is in the range 0.4 to 0.9.

Said method preferably comprises selecting a said first polymeric material; selecting a second polymeric material which includes a functional group which is able to react in the presence of said first polymeric material to form said third polymeric material; and causing the formation of said third polymeric material by a reaction involving said first and second polymeric materials.

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The ratio of the wt% of said first polymeric material to the wt% of said second polymeric material selected for preparation of said third polymeric material is suitably less than 0.15, preferably less than 0.10, more preferably less than 0.05, especially less than 0.035. Said ratio may be at least 0.005, preferably at least 0.01, more preferably at least 0.01, especially at least 0.02.

The sum of the wt% of the first and second polymeric materials selected for preparation of said third polymeric material may be at least 0.4 wt%. The sum may be less than 5 wt%, preferably less than 4 wt%, more preferably less than 3 wt%, especially less than 2.5 wt%.

30 Suitably, the amounts of "first polymeric material" and "second polymeric material" described refer to the sum of the amounts of first polymeric materials (if more than one

type is provided) and the sum of the amounts of second polymeric materials (if more than one type is provided).

Water for use in the treatment fluid formulation may be 5 derived from any convenient source. It may be potable water, surface water, sea water, aquifer water, deionised production water and filtered water derived from any of The water may be treated so the aforementioned sources. that it is suitable for use in the method. For example, 10 it may be treated by addition of oxygen scavengers, biocides, corrosion inhibitors, scale inhibitors, antifoaming agents and flow improvers. Sea water and/or water and/or be deoxygenated other sources may from desulphonated.

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In the preparation of said third polymeric material a catalyst is preferably provided for catalysing the reaction of the first and second polymeric materials. Said catalyst is preferably a protic acid. Said catalyst is preferably phosphoric acid. Advantageously, when the fluid flow path is defined by steel pipes the phosphoric acid may facilitate the formation of an anti-corrosive layer on the pipes.

In the materials described above, A and/or B could be . 25 heteroaromatic multi-cyclic aromatic or Preferably, A and B are independently selected from optionally-substituted five more preferably or membered aromatic and heteroaromatic groups. Preferred heteroatoms of said heteroaromatic groups 30 nitrogen, oxygen and sulphur atoms of which oxygen and preferred. Preferred especially nitrogen, are heteroaromatic groups include only one heteroatom.

preferably, a or said heteroatom is positioned furthest away from the position of attachment of the heteroaromatic group to the polymer backbone. For example, where the heteroaromatic group comprises a six-membered ring, the heteroatom is preferably provided at the 4-position relative to the position of the bond of the ring with the polymeric backbone.

Preferably, A and B represent different groups.

Preferably, one of A or B represents an optionallysubstituted aromatic group and the other one represents an
optionally-substituted heteroaromatic group. Preferably A
represents an optionally-substituted aromatic group and B
represents an optionally-substituted heteroaromatic group
especially one including a nitrogen heteroatom such as a
pyridinyl group.

Unless otherwise stated, optionally-substituted groups described herein, for; example groups A and B, may be substituted by halogen atoms, and optionally substituted 20 hemiacetal, acetalalkyloxy. acyl, acetal, hemiacetalalkyloxy, nitro, cyano, alkoxy, hydroxy, amino, alkylamino, sulphinyl, alkylsulphinyl, sulphonyl, alkylsulphonyl, sulphonate, amido, alkylamido, alkylcarbonyl, alkoxycarbonyl, halocarbonyl and haloalkyl 25 Preferably, up to 3, more preferably up to 1 optional substituents may be provided on an optionally substituted group.

30 Unless otherwise stated, an alkyl group may have up to 10, preferably up to 6, more preferably up to 4 carbon atoms, with methyl and ethyl groups being especially preferred.

Preferably, A and B each represent polar atoms or group - that is, there is preferably some charge separation in groups A and B and/or groups A and B do not include carbon and hydrogen atoms only.

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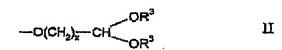
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Preferably, at least one of A or B includes a functional group which can undergo a condensation reaction, for example on reaction with said second polymeric material. Freferably, A includes a said functional group which can undergo a condensation reaction.

preferably, one of groups A and B includes an optional substituent which includes a carbonyl or acetal group with a formyl group being especially preferred. The other one of groups A and B may include an optional substituent which is an alkyl group, with an optionally substituted, preferably unsubstituted, C<sub>1-4</sub> alkyl group, for example a methyl group, being especially preferred.

20 Preferably, A represents a group, for example an aromatic group, especially a phenyl group, substituted (preferably at the 4-position relative to polymeric backbone when A represents an optionally-substituted phenyl group) by a formyl group or a group of general formula

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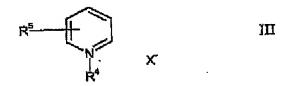


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where x is an integer from 1 to 6 and each  $R^3$  is independently an alkyl or phenyl group or together form an alkalene group.

5 Preferably, B represents an optionally-substituted heteroaromatic group, especially a nitrogen-containing heteraromatic group, substituted on the heteroatom with a hydrogen atom or an alkyl or aralkyl group. More preferably, B represents a group of general formula

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wherein  $\mathbb{R}^4$  represents a hydrogen atom or an alkyl or aralkyl group,  $\mathbb{R}^5$  represents a hydrogen atom or an alkyl group and X represents a strongly acidic ion.

Preferably, R<sup>1</sup> and R<sup>2</sup> are independently selected from a hydrogen atom or an optionally-substituted, preferably unsubstituted, alkyl group. Preferably, R<sup>1</sup> and R<sup>2</sup> represent the same atom or group. Preferably, R<sup>1</sup> and R<sup>2</sup> represent a hydrogen atom.

25 Preferred first polymeric materials may be prepared from any of the compounds described on page 3 line 8 to line 39 of GB2030575B by the method described in WO98/12239 and the contents of the aforementioned documents are incorporated herein by reference.

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Said first polymeric material may be of formula

wherein A, B,  $R^1$  and  $R^2$  are as described above and n is an integer. Integer n is suitably 10 or less, preferably 8 or less, more preferably 6 or less, especially 5 or less. Integer n is suitably at least 1, preferably at least 2, more preferably at least 3. Preferably, formation of said third polymeric material from said first and second 10 polymeric materials involves a condensation reaction. Preferably, formation of said third polymeric material involves an acid catalysed reaction. Preferably. first and second polymeric materials include functional groups which are arranged to react, for example to undergo a condensation reaction, thereby to form said third Preferably, said first and second polymeric material. polymeric materials include functional groups which are for example to undergo arranged to react catalysted reaction thereby to form said third polymeric 20 material.

Preferably, said second polymeric material includes a functional group selected from an alcohol, carboxylic acid. carboxylic acid derivative, for example an ester, and an Said second polymeric material preferably amine group. preferably consisting . includes a backbone comprising, essentially of carbon atoms. The backbone is preferably Pendent from the backbone are one or more said saturated. functional groups described. Said polymer may have a number average molecular weight (Mn) of at least 10,000, preferably at least 50,000, especially at least 75,000. may be less than 500,000, preferably less than 400,000. Said second polymeric material is preferably a polyvinyl

polymer. Preferred second polymeric compounds include optionally substituted, preferably unsubstituted, polyvinylalcohol, polyvinylacetate, polyalkylene glycols, for example polypropylene glycol, and collagen (and any component thereof) and of these polyvinylalcohol and/or polyvinylacetate based polymeric materials are preferred.

Preferably, said second polymeric material includes at least one vinyl alcohol/vinyl acetate copolymer which suitably includes greater than 50%, preferably greater than 65%, more preferably greater than 80wt% of vinyl alcohol moieties. Where two types of second polymeric material are provided both may be vinyl alcohol/vinyl acetate copolymers Preferably, said copolymers are of the type described. substantially identical except for differences in their number average molecular weights. The provision of two types of second polymeric materials of different properties (e.g. number average molecular weight) provides a means whereby the viscosity of a formulation of the first and second materials before reaction to form the polymeric material may be adjusted.

Said third polymeric material suitably includes a moiety of formula

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1.0

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wherein  $R^1$ ,  $R^2$  and B are as described above,  $A^1$  represents a residue of group A described above after the reaction involving said first and second polymeric materials, Y represents a residue of said second polymeric material after said reaction involving said first and second

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polymeric materials and X represents a linking atom or group extending between the residues of said first and second polymeric materials. In one preferred embodiment A<sup>1</sup> represents an optionally-substituted phenyl group, X represents a group



which is bonded via the oxygen atoms to a residue of said second polymeric material. For example, group X may be bonded to the polymer backbone of said second polymeric material.

The method of the first aspect preferably includes the step of contacting the viscous composition after it has been contacted with said treatment fluid formulation with a breaker means arranged to break an aqueous emulsion of the viscous composition. The breaker means way contacted with the other materials after emulsion of the viscous composition has been transported to a desired location. When the second polymeric material comprises polyvinylalcohol. the breaker preferably arranged to cleave 1,2-diol linkages of the polyvinylalcohol. Said breaker means preferably comprises a periodate (e.g. sodium or potassium periodate) in water.

According to a second aspect of the present invention there is provided a method of preparing a treatment fluid formulation (e.g. for reducing the viscosity of a viscous composition) comprising:

selecting a first polymeric material and a second polymeric material as described according to said first aspect; and

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causing the formation of a said third polymeric material by a reaction involving said first and second polymeric materials.

The method may include contacting the first and second polymeric materials with an acid for catalysing the reaction thereof.

Preferably, at least 100 litres of said treatment fluid formulation is prepared.

According to a third aspect of the invention, there is provided a treatment fluid formulation comprising:

- 20 at least 95wt% water
  - 4wt% or less of said third polymeric material described above.

The invention extends to a receptacle containing at least 100 litres, preferably at least 200 litres, especially at least 1000 litres of a said treatment fluid formulation according to said third aspect.

According to a fourth aspect of the invention, there is provided a fluid flow path, for example a conduit means (preferably having a cross-sectional diameter at least in part of at least 5cm and a length of at least 5m) which

contains a fluid comprising petroleum, water and a third polymeric material as described herein.

Any feature of any aspect of any invention or embodiment described herein may be combined with any feature of any aspect of any other invention or embodiment described herein mutatis mutandis.

Specific embodiments of the invention will now be

10 described, by way of example, with reference to figure 1

which is a plot of viscosity vs. shear rate for various

formulations.

general terms, heavy crude oil (and associated material) which may be too viscous to enable it to be pumped from the flowing face of a reservoir into and along a pipeline, for example to a refinery or other storage facility, may be contacted with a hydrogel formulation at any point where it is desirable to reduce the oil viscosity. The hydrogel is formed by reaction of 20 poly(1,4-di(4-N-methylpyridinyl))-2,3-di(4-(1formylphenyl)butylidene and poly(vinyl alcohol) presence of an acid catalyst. It is formulated as an acueous formulation which is adapted to reduce 25 viscosity of the oil thereby to facilitate transportation. The viscosity may be reduced by the hydrogel formulation emulsifying the oil. When desired, the emulsified oil may be broken to enable the oil to be separated from the hydrogel so the oil may be used as

Further details on the process are provided below.

desired.

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# Example 1 - Preparation of poly (1,4-di(4-(N-methylpyridinyl))-2,3-di(4-(1-formylphenyl)butylidene

of Example described in as prepared This was PCT/GB97/02529, the contents of which are incorporated herein by reference. In the method, an aqueous solution of 4-(4-formylphenylethenyl)-1οĒ WES than greater is prepared by methylpyridinium methosulphonate (SbQ) mixing the SbQ with water at ambient temperature. Under such conditions, the SbQ molecules form aggregates. The solution was then exposed to ultraviolet light. This results in a photochemical reaction between the carboncarbon double bonds of adjacent 4-(4-formylphenylethenyl)-1-methylpyridinium methosulphate molecules **(1)** poly (1,4-di(4-(Npolymer, producing a aggregate, 15 methylpyridinyl))-2,3-di(4-(1-formylphenyl)butylidene methosulphonate (II), as shown in the reaction scheme It should be appreciated that the anions of compounds I and II have been omitted in the interests of clarity. 20

>1%w/w Aqueous solution UV irradiation

# 5 Example 2 - Preparation of poly(vinylalcohol) solution

A 10wt% poly(vinylalcohol) solution was prepared by slowly stirring a known amount of water and adding a known amount of 88% hydrolysed poly(vinylalcohol) to the stirred water. The suspension was stirred for 1 hour and, thereafter, the suspension was heated at a temperature of 60°C until the suspended particles dissolved and the solution was clear.

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The solution was then allowed to cool to less than 5°C and maintained at this temperature until used.

Example 3 - Preparation of butylidene polymer/poly(vinylalcohol) formulation

997.5g of the poly(vinylalcohol) solution prepared Example 2 and 2.5g of the butylidene polymer prepared in Example 1 were mixed together at ambient temperature to poly(vinylalcohol)/0.25wt% butylidene wta 10 10 This was diluted down to give a 2wt% polymer solution. poly(vinyl alcohol)/0.05wt% butylidene polymer solution. This solution was acidified to pH 1.5 with phosphoric acid After curing the solution and left to cure for 1 hour. This cured and neutralised was neutralised using 5M NaOH. 15 solution was then further diluted to give a poly(vinylalcohol)/0.025wt% butylidene polymer solution; 0.05wt% polyvinylalcohol/0.0125wt% and polymer solutions.

On acidification of the polyvinylalcohol/butylidene polymer blend as described, the two polymers react according to the scheme below:

Example 4 - Protocol for preparation of cil emulsions

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Emulsions of oil and the aqueous formulations of Example 3 were prepared at ratios of oil: aqueous formulation of 70:30 and 60:40 with the aqueous phase containing 2wt%, lwt% or 0.5wt% poly(vinylalcohol). The oil and aqueous formulations were initially mixed using a spatula, then homogenized.

## Example 5 - Protocol for evaluation of oil emulsions

The viscosity against shear rate of the emulsions was measured on 0.65ml samples at 25°C taking 60 measuring points at 10 second intervals and a shear rate of 0.1 to 1000s<sup>-1</sup> followed immediately by a rate of 1000 to 0.1s<sup>-1</sup>. Results are provided in Figure 1 wherein:

- Lines A and A<sup>1</sup> are comparative examples detailing the results for two runs undertaken without inclusion of an aqueous formulation of Example 2.
- 20 Lines B and B' are the results for two runs undertaken using the aqueous formulation of Example 3 with 2wt% poly(vinylalcohol).
  - Lines C and  $C^1$  are the results for two runs undertaken using the aqueous formulation of Example 3 with 1wt% of poly(vinylalcohol).
  - Lines D and D1 are the results for two runs undertaken using the aqueous formulation of Example 3 with 0.5wt% of polyvinylalcohol.
- Figure 1 shows that without the addition of any formulation described in Example 3, the viscosity is relatively high. When the formulation is added, there is a significant reduction in viscosity.

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Thus, the formulation may be dosed into a cil flow at any point at which it is desired to reduce the cil's viscosity to enable it to be transported. For example, it may be closed in at the bottom of a riser pipe to reduce the viscosity of oil flowing upwardly in the pipe. Alternatively, it may be closed in at or near the surface. Once closed in, the oil may be transported long distances through a pipeline to a refinery or other oil storage facility.

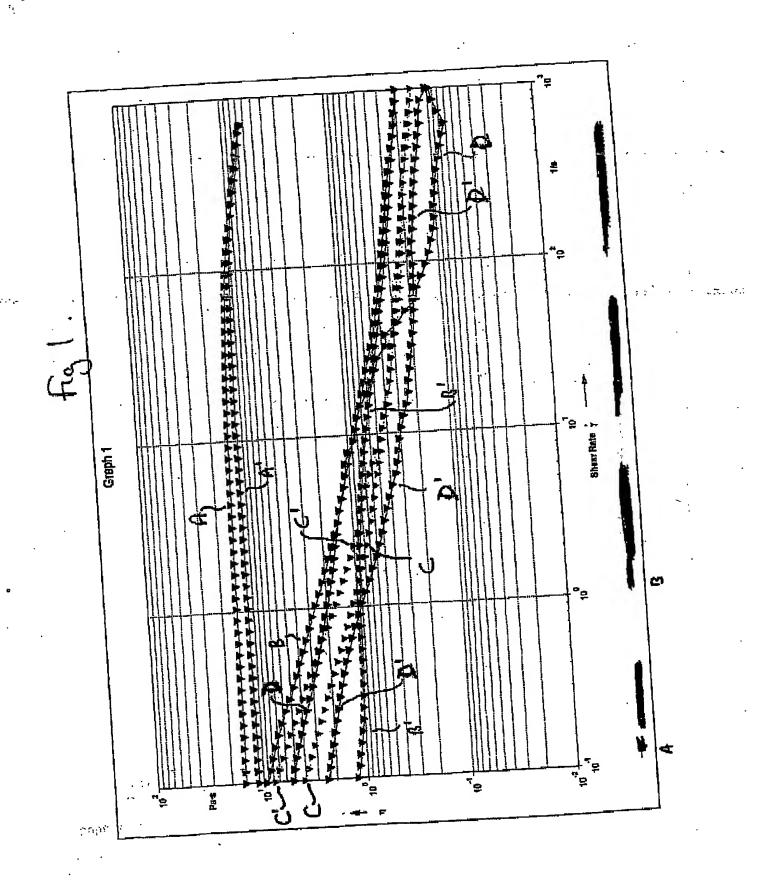
After completion of the transport stage, it is necessary to recover the oil from the emulsion. This is achieved by the addition of 0.1 to 0.3wt% (preferably about 0.2 wt%) of a periodate salt (preferably the sodium salt) to the emulsion. This causes the destruction of the emulsion and enables the oil to be recovered for further processing.

Attention is directed to all papers and documents which are filed concurrently with or previous to this specification in connection with this application and which are open to public inspection with this specification, and the contents of all such papers and documents are incorporated herein by reference.

All of the features disclosed in this specification (including any accompanying claims, abstract and drawings), and/or all of the steps of any method or process so disclosed, may be combined in any combination, except combinations where at least some of such features and/or steps are mutually exclusive.

Each feature disclosed in this specification (including any accompanying claims, abstract and drawings) may be replaced by alternative features serving the same, equivalent or similar purpose, unless expressly stated otherwise. Thus, unless expressly stated otherwise, each feature disclosed is one example only of a generic series of equivalent or similar features.

The invention is not restricted to the details of the foregoing embodiment(s). The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any method or process so disclosed.



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